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The applicability of gecko-inspired dry adhesives to the conservation of photographic prints

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ABSTRACT

The application of gecko-inspired dry adhesives (GDAs) for conservation treatments on photographic prints is presented. GDAs attach to a substrate surface with a row of micro-pillars. This architecture, which is designed to replicate gecko feet and hence promote van der Waals forces, results in strong adhesion in the normal and shear directions under tension, but low peel strength, offering the potential of ease of removal from fragile surfaces during treatment, and use as a mounting adhesive. The adhesion properties of Gecko Nanoplast were assessed on the print side of contemporary photographic paper using tensile, shear and peel tests. A maximum shear adhesion force of 48.1 N and peel force of 0.4 N confirmed the strong adhesion to the prints, combined with ease of detachment without residue. The micro-pillar structure of the GDA resulted in peel and shear behaviour that is indicative of Van der Walls forces acting as the adhesion force.

INTRODUCTION

Sixty percent of geckos have the ability to walk on the majority of surfaces, regardless of the angle between the surface and the ground. These geckos have rows of ‘nanohairs’ on the underneath of their feet called ‘setae’ (about 3–4 µm in diameter and 30–130 µm in length). The setae branch into even smaller hairs on the very top called ‘spatulae’, with dimensions of approximately 5 × 150 nm. Because of their scale, the spatulae have very close proximity to the atoms on the surface of a material so that adhesion occurs via van der Waals secondary bonding forces (Putthof et al. 2013). A review of gecko adhesion mechanisms in live animals outlines and discusses the theories behind gecko adhesion (Autumn et al. 2014).

GDAs do not require heat or solvents for curing, and no apparent adhesive migration or chemical interaction occurs with the adherend surfaces. The resulting adhesive bond has relatively high adhesive shear strength and low peel strength, leaving a surface without residue. Therefore, GDAs seem to be very promising materials for remedial and permanent conservation applications.

The use of gecko adhesives in the conservation of art was first suggested in 2011 (Fenn 2011); possible applications included mounting systems for showcases and emergency aid in cases of damage. To the authors’ knowledge, there are no other published results within the conservation literature with the exception of Izadi et al. (2016), who proposed a method of removing surface dirt from smooth surfaces with a nanofibrillar-surfaced polymer.

GDA MANUFACTURING AND TESTING

Research on synthetic GDAs has focused on their evaluation for applications in robotics or the electronics industry. GDAs are produced commercially by a handful of companies (mostly university spin-offs; see Table 4). Each new type of GDA is tested for different parameters and with different methods. To date, the manufacturers have yet to establish a common set of testing protocols. GDAs are manufactured using either carbon nanotubes, UV-etching, nano-moulding, micro- and nano-carving. Each method requires different materials and preparation procedures, resulting in different final properties, e.g. adhesive force, material rigidity, anisotropy of the adhesion, and longevity. Testing and manufacturing methods have been reviewed in important studies that include details on the methods of fabrication, achieved adhesion properties, resistance to multiple reattachments and test methods (del Campo and Arzt 2007, Jeong and Suh 2009). A review of the numerous methods of testing GDA performance (e.g. nano-indentation, atomic force microscopy, laboratory balances) points out that a universal testing protocol does not exist for fibrillar surfaces-based adhesives at the present time (Boesel 2010).

To assess the performance of GDAs made of vertically aligned carbon nanotubes on glass, tack and shear tests were carried out (Chen et al. 2015). However, the shear test was prepared by gluing a wire to the back of the sample, which may have caused significant unevenness of force distribution during testing. A similar approach has been taken by King et al. (2014) to evaluate GDA performance on 'real life' materials – wood panels or painted drywall. The samples tested were scaled to 'real life' applications, mounted with a preload and pulled at 0° and 90° angles. A load-drag-pull test has also been employed to evaluate GDAs made of Teflon AF nanopillars; the method was based on attaching a semi-spherical probe to the adhesive material with a pre-set preload, pulling it along the sample at a constant speed, and finally detaching the probe perpendicularly to the material surface at a constant speed. The method combines tack and shear tests together and involves dynamic changes, more akin the action of a gecko (Izadi 2014).

The industry standards for mechanical testing of adhesives (ASTM, ISO, European Union and PSTC)[1] describe the methodology for properties including: 1) tack, the ability of the adhesive to create an immediate bond with a substrate, usually measured in the normal direction; 2) normal adhesion, measured as a tension perpendicular to adhesive, after the adhesive has cured; and 3) peel, a force localised at the line of detachment of the adhesive (Brockmann et al. 2009). However, conservation research on adhesives has not always used standardised testing methods; this is in part because they are derived for industrial applications that cannot be directly translated to most of the conservation procedures. The Canadian Conservation Institute studies of adhesives between 1982 and 2014 (CCI 2016) focused on 27 poly(vinyl acetate) and 25 acrylic adhesives comparing compositional purity, acidity and alkalinity (of both basic products and cured films), volatile compounds emission, yellowing and cohesive strength (Down et al. 1996, Down 2009). Mechanical tests were limited to the tensile tests of cured films, in compliance with ASTM standard D2370-82, from which the stress and strain were calculated. No adhesive force was measured to test the interaction performance of the adhesive with different substrate surfaces.

Peel testing has regularly been used to compare adhesives between two materials in conservation applications (Young et al. 2002). Pressure-sensitive acrylic esters used as lining adhesives have been compared using standardised peel tests (ASTM D903-49) before and after artificial ageing (Roche 1996). Uni- and biaxial tensile tests have also been performed to compare the performance of linings for canvas paintings (Ackroyd and

Young 1999). Thuer (2010) made a comparative assessment of adhesives to develop the methodology of facings preparation for glue-tempera painted wood using a non-standardised 180° peel test on a stiff substrate.

The strength of an adhesive joint depends on the properties of the adhesive and on the properties of the adherends. Therefore, the research methodology in this project involves characterisation of both joint strength and adherends. This research is aimed at providing future conservators with a general idea of what might be expected from this new type of adhesive; thus, assessing all the factors that could influence the final joint strength is crucial. Adhesive testing methodologies need development for GDAs because, conventionally, adhesive materials have two adherends joined to the adhesive. For pressure-sensitive adhesive tapes and labels, the second adherend is simply the backing material of the tape or label. But in the case of GDAs, the adhesive is also the backing material; ergo, it is an adherend as well.

In the preliminary stages of this research, the authors adhered Gecko Nanoplast to 19th-century linen canvas with and without paint layers to assess its application as part of a facing system. However, the results showed that the viscoelastic backing material of the Gecko Nanoplast gave a better bond than the actual 'gecko' surface. The 'tack' due to a low glass transition temperature (Tg) of the substrate (see below) rather than van der Waals forces provided a good bond in this case. The reverse side of the Gecko Nanoplast also performed well as an adhesive in the realignment of canvas paintings during tear mending. Therefore, the research at this stage has now focused on photographic prints because they are relatively stiff, elastic materials with a very smooth surface. The preliminary results suggest that these properties are most likely to succeed for this particular GDA used on the front side (Young and Olender 2016). After consultations with paper conservators with considerable experience of photographic prints, the use of a GDA as a temporary connection system for damaged photographs during the process of reassembling torn prints (that would allow the photographs to be turned in order to adhere them together from the verso) was experimentally assessed. After empirical tests with Gecko Nanoplast in photographic prints originating from the 1960s–1990s, contemporary commercial prints with a glossy surface were selected. Although different to archival prints, these allowed for consistency, in addition to being considered one of the most problematic surfaces in photography conservation (Messier 2016, Townshend 2016).

EXPERIMENTAL METHODS AND RESULTS

The only over-the-counter commercially available GDA, Gecko Nanoplast, was obtained from Kletteband Technik (dealer of Gottlieb Binder products) as a 25 × 10 cm sheet. Patents pending have prevented the assessment of other GDAs at this stage. Photographic prints, with dimensions of 15.2 × 10.2 cm, all copies of one image, were ordered at a commercial photographic laboratory. Prints were prepared from a digital file, with glossy finish and without any size adjustments.

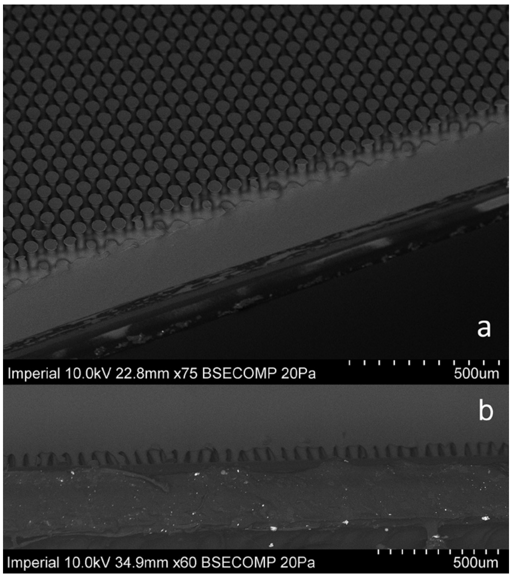

Figure 1. ESEM images of Gecko Nanoplast: (a) angled view; (b) cross-sectional view

Optical microscopy and environmental scanning electron microscopy (ESEM)

Samples were imaged under ESEM at 10 KeV (SEM images showed that the metal coating required flattened the 'gecko' pillars). The ESEM image in Figure 1a shows the GDA architecture. Figure 1b shows two layers separated with a wave-shaped border just below the 'gecko' pillars which are about 50 μm high and are positioned about 50 μm centre-to-centre from each other.

Fourier transform infrared spectroscopy – Attenuated total reflectance spectroscopy (FTIR-ATR)

The Gecko Nanoplast composition was characterised with FTIR-ATR. Samples of 50 × 20 mm in size were placed on the scanning crystal and measured on both sides. The FTIR results compared to reference data show that the substrate is polyurethane, and the 'gecko' side is made of poly(dimethyl siloxane) (PDMS).

Dynamical mechanical analysis

Gecko Nanoplast was measured using dynamical mechanical analysis (DMA) to obtain its glass transition temperature (Tg) profile. A sample with dimensions of 14.79 × 5.49 × 0.42 mm (l/w/t) was tested in tension at a 1Hz frequency and a temperature change from -150°C to 35°C, with a 3°C/min change rate. As expected, a double peak was seen in the tan delta curve (see Figure 2), corresponding to published data for the Tg of PDMS and polyurethane elastomers – approximately 125°C (Lötters et al. 1999) and -20°C, respectively (BASF 2016). This measurement confirmed the FTIR data on the chemical composition of the material and its composite nature. Polyurethane does not have good ageing properties and thus another substrate would be sort for long-term conservation applications. The low Tg for PDMS confirms its tackiness and potential to creep.

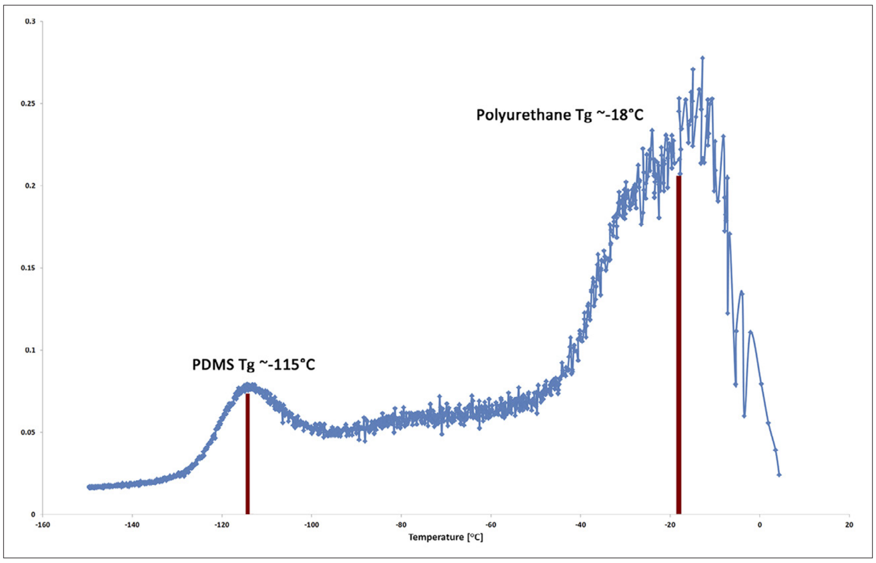

Figure 2. Tan delta graph of Gecko Nanoplast from the DMA test

Tensile tests

Because the adherends are viscoelastic and have flexible peel arms it is necessary to take into account the extension they will undergo during peel and shear testing. This also allows the adhesive fracture energy to be calculated and compared to published data. Tensile tests were conducted using an Instron 4301 universal tester with an in-house integrated controlled environmental chamber. The conditions in the chamber were 20°C ± 1°C and 33% RH ± 3%. Testing conditions meet the British Library guidelines for storage of archival materials (Henderson 2013). A 1000 N load cell was used and the cross head speed was 10 mm/min.

Gecko Nanoplast

Five samples were prepared (9 cm long and 2 cm wide) with a gauge length of 7 cm. The thickness of the sample was measured in three positions and the average value was calculated. The material was supplied covered with a protective film on each side. The technical specification suggests that the material should not show anisotropic behaviour. It was not possible to measure the thickness of the material directly because it would stick to the micrometer. The samples were measured with the protective films, after which the films were measured separately and their thickness was subtracted from the overall measurement.

Samples of Gecko Nanoplast were tensioned without yield or failure to a maximum strain of 71.3% ± σ 0.1% (50 mm extension) and average maximum stress of 101.9 MPa ± σ 4.1 MPa (see Figure 3 and Table 1).

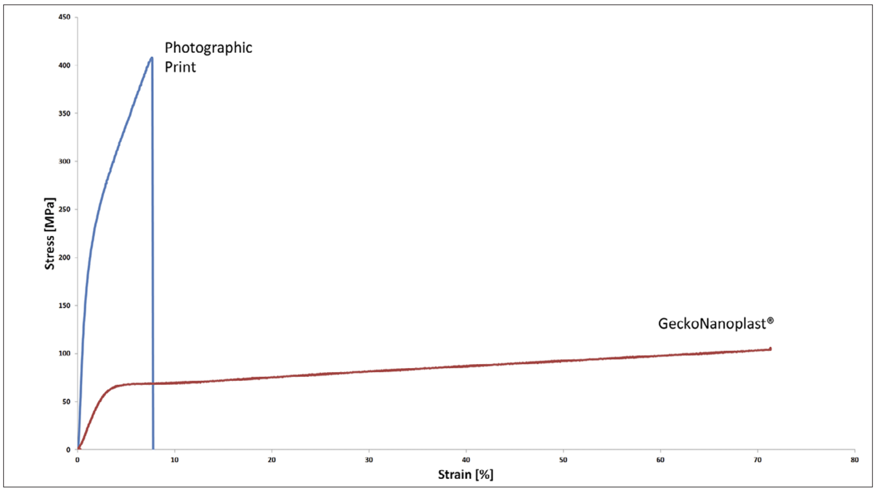

Figure 3. (a) Strain vs strain graph of Gecko Nanoplast and the photographic print

Table 1. Summary of tensile tests of Gecko Nanoplast and photographic print

Tensile tests summary								
	Gecko® Nanoplast®				Photographic prints			
Sample nr.	Max. force [N]	Extension at yield	Max. stress [MPa]	Strain [%]	Max. force [N]	Extension at break	Max. stress [MPa]	Strain [%]
1.	76.5	2.3	97.3	71.3	167.8	5.4	407.9	7.7
2.	80.9	2.2	104.7	71.3	168.3	5.4	410.5	7.8
3.	79.7	1.9	105.9	71.3	171.2	5.4	419.0	7.8
4.	74.5	2.2	99.8	71.3	171.1	5.7	418.7	8.1
5.	--	--	--	--	164.5	5.3	401.8	7.6
Average	77.9	2.1	101.9	71.3	168.6	5.4	407.9	7.8
σ±	2.9	0.1	4.0	0.0	2.8	0.1	7.3	0.2

Photographic paper

Five samples where cut from the prints and the thickness measured in three positions in order to calculate average thickness. Testing conditions were 35% ± 3% RH and 20°C ± 1°C. The average maximum strain was 7.8% ± σ 0.2% at an average tensile stress of 411.6 MPa ± σ 7.3 MPa (see Figure 3 and Table 1).

Shear tests

Strips (9 cm long and 2 cm wide) were prepared with an overlap of 4 cm, leaving 2.5 cm at each end. The gauge length between the grips was 7 cm. The Gecko Nanoplast was applied onto the face of the photographic print and pressed with a finger through the layer of protective film, which was removed after the thickness measurements. Thickness was measured in three positions on the overlap. The tests were conducted in 35% ± 2% RH and 20°C ± 1°C. The average maximum shear force was 48.1 N ± σ 2.7 N, at an average extension of 1.6 mm ± σ 0.2 mm. However, an intriguing phenomenon was observed in the later part of the test. In every sample, after reaching a peak force, the adherends did not separate. In two cases, adhesion dropped to 4.5–5.5 N almost immediately reaching again 12–13 N, and dropping again to 8 N. In the other three cases, adhesion fell straight to 8–10 N. However, in all five samples the GDA remained adhered and the force was measured until it slid off the photographic paper. An average value of 8.6 N ± σ 1.5 N was measured at the beginning of the slope, and the adhesion fell at an average rate of 0.2 N/mm ± σ 0.1 N/mm. The slope is characterised by a series of force peaks, which suggests the presence of the effect of micro-sliding of the 'gecko' pillars (see Figures 4 and 5).

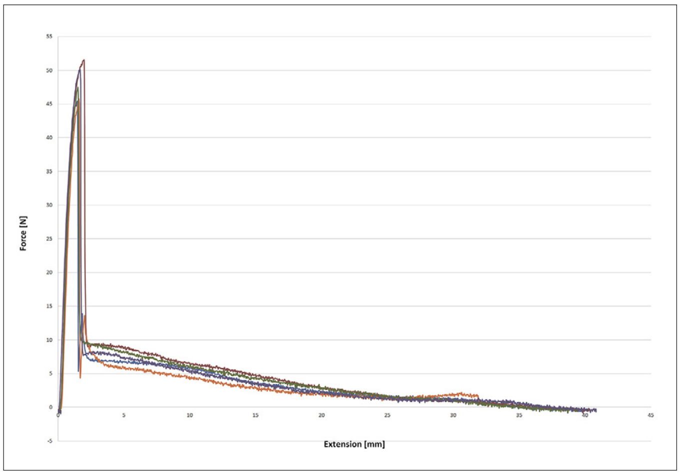

Figure 4. Load vs extension of tensile shear data

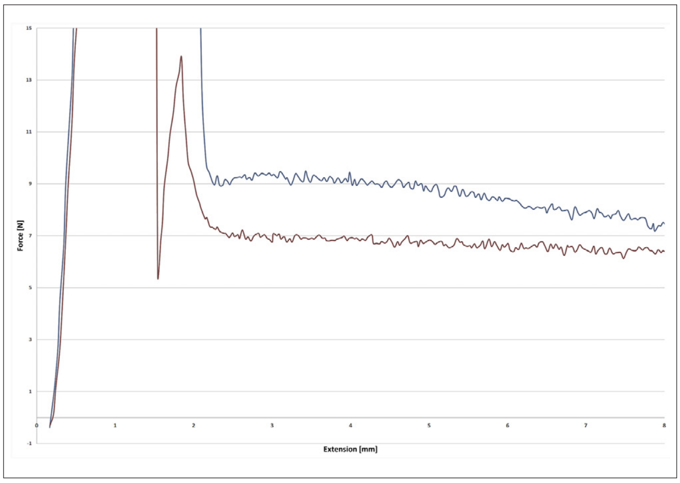

Figure 5. Detail of the shear force strength of two samples, one showing the extra force peak and one without this phenomenon. Wavy force slope is visible as well

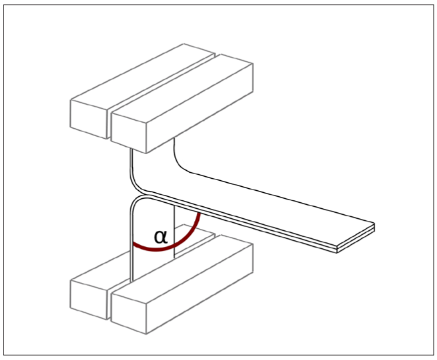

Figure 6. Schematic diagram of 180° peel test. Peel angle marked with 'α'

Table 2. Summary of peel test results

Peel forces summary		
Sample nr.	Average peel force [N]	Std. deviation [N]
1.	0.5 N	0.1 N
2.	0.5 N	1.2 N
3.	0.4 N	0.1 N
4.	0.1 N	0.1 N
5.	0.7 N	0.1 N
Average	0.4 N	N.A.
Std. deviation	0.2 N	N.A.

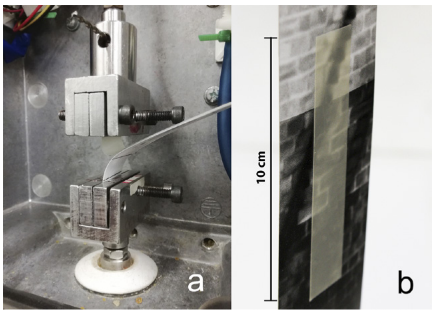

Figure 8. (a) Peel-off sample set in the tensile tester prepared for the procedure; (b) a sample of Gecko Nanoplast using gecko adhesion to stick to a vertically hanging photographic print

Peel tests

The 180° peel strips were 9 cm long and 2 cm wide with the adherends connected along a 7-cm-long section; 1 cm on each of the adherends was left for clamping in the Instron grips (see Figures 6 and 8a). Five tests were conducted in 35% ± 2% RH and 20°C ± 1°C. From each sample, 30 mm of stable, continuous peel strength data was selected from which the average force was calculated (Kinloch et al. 1994, Moore and Williams 2010). The average force for the five samples was 0.4 N, with a range between 0.1 N and 0.7 N (see Figures 7a and 7b, and Table 2). Because of the difference in adherend stiffness, the angle between the bottom adherend (photographic print) and the free end of the sample measured during the test was between 156 and 160°. The amplitude of the peaks and troughs of the peel section is indicative of cohesive failure. However, no residue of GDA is left on the adherend. Together with the shear data, this suggests that the 'gecko' pillars are being separated from the surface as the peel continues.

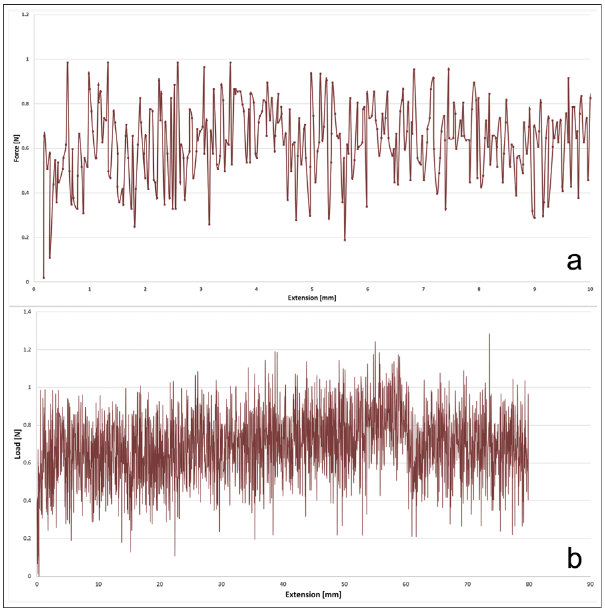

Figure 7. Load vs extension data: (a) complete peel test, (b) first 10 mm of the peel test

DISCUSSION AND CONCLUSION

The adhesive bond in the tensile shear direction is strong, while the peel force is extremely low. The nature of the detachment of Gecko Nanoplast from the print surface suggests that a 'gecko' van der Waals-type adhesion is taking place. Visual assessment of the surfaces of the photographic samples did not show any damage or residue. The continuing research includes tests at a range of RH and temperature, shear creep tests and ESEM examination of different photographic print surfaces before and after testing.

The results have shown that gecko-inspired dry adhesives have potential to become useful conservation materials. Until data on the long-term performance can be acquired, only short-time use can be regarded as safe (e.g. immobilising fragments of photographic prints, see Figure 8b). The use of this particular GDA as a way of creating temporary connections between fragments of photographs (or similar flat surfaces) could be considered a valid method for conservators.

Table 3. Summary of shear test results; two last columns describe the 're-attachment effect'

Shear tests summary				
Sample nr.	Max. force [N]	Extension at F_{max} [mm]	Re-attachment force [N]	Force decrease [N/mm]
1.	45.4	1.5	8.6	0.2
2.	51.5	2.0	9.7	0.3
3.	45.8	1.6	6.1	0.2
4.	47.5	1.5	9.9	0.3
5.	50.1	1.7	8.5	0.2
Average	48.1	1.6	8.5	0.2
Std. dev.	2.7	0.2	1.5	0.0

Table 4. List of GDA manufacturers

Gecko-inspired dry adhesives manufacturers		
Company name	**Country**	**Product name**
Gottlieb Binder GmbH	Germany	Gecko Nanoplast®
Phelsuma Inc.	USA	Geckskin®
nanoGriptech	USA	Setex™
Leibniz-Institut für neue Materialen	Germany	Gecomer®

Table 5. List of instruments used

List of instruments	
Type of test/analysis	**Instrument manufacturer and model**
Optical microscopy	Leica DM 4000 M LED with digital camera Leica DFC 450C
ESEM	Hitachi S-3400 N Variable Pressure SEM
DMA	TA Instruments Q800 v21.1
FTIR-ATR spectroscopy	Bruker ALPHA with Platinum ATR module
Tensile/shear/peel tests	Instron 4301 Universal tester

NOTES

1 ASTM – organisation originally founded as the American Section of the International Association for Testing Materials, now known as ASTM International; ISO – International Organization for Standardization, an external organisation providing industrial standards; PSTC – Pressure Sensitive Tape Council, a trade association for pressure-sensitive adhesive manufacturers.

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